CHARACTERIZATION AND GENETIC STUDY OF A TYPICAL PRAIRIE AND A TYPICAL PLANOSOL OF EASTERN OKLAHOMA

By

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INTRODUCTION

It is apparent that knowledge of the clay mineral constituents of soils is becoming increasingly important. Many studies have been conducted in recent years to determine the effects of specific mineral groups on the physical and chemical properties of soils and, ultimately, their effect on plant growth.

A knowledge of the kinds and amounts of clay minerals present in a soil which presents agronomic problems is helpful in making recommendations for most effective and efficient use of that soil. The genetic relationships which exist between the mineral in soil profiles and those in soil parent materials aid in the characterization of soils and contribute to more sound soil classification.

Dennis and Parsons soils occupy extensive acreage in eastern Oklahoma and adjoining states. They are agronomically important and are intensively farmed. The Dennis series is representative of the Prairie Great Soil Group while the Parsons is a typical Planosol. The two soil series are associated geographically and are related genetically. They are developed from similar parent material but exist in different stages of morphological development.

The purposes of this study were: (1) to identify the kinds and amounts of clay minerals present in each horizon of the two soil series, Dennis and Parsons, and (2) to evaluate the clay mineral contents of the genetic horizons of both soil profiles as keys to morphological development within the respective soil profiles and as keys to the genetic relationships which exist between the two soil series.

REVIEW OF LITERATURE

Methods of Analysis

The clay minerals are of fundamental importance in the soil. Since their recognition as crystalline materials (17, 29), many detailed investigations have been conducted to determine the specific types and amounts of minerals present in soils. The common clay minerals in soils are classified into five broad groups as follows: (1) The montmorillonite group, (2) kaolinite, (3) illite, (4) vermiculite, and (5) the mixed layered minerals.

Success in proper identification and estimation of amounts of clay minerals is dependent upon the investigator's knowledge of the structures of clay minerals and the methods he used to identify these structures.

Cation exchange capacity, ethylene glycol retention, analysis of chemical constituents, differential thermal analysis, X-ray spectrographic analysis, dehydration curves, dye adsorption, optical properties and electron microscope examination have been used for identifying clay minerals.

Unless only one clay mineral is present in a soil clay, two or more of the given methods are necessary for identification of the minerals in a soil clay.

The principal involved in the methods of analyses used are as follows:

X-ray Analysis

When an X-ray beam of length sufficient to pass through the structure of a material, is beamed through a crystalline material, it is diffracted upon striking any plane surface. Kelley, Dore and Brown (29) and Hendricks and Fry (17), using X-ray equipment, established that soil colloids, formerly thought to be amorphous, were crystalline materials. Other investigations, (46, 17, 33), have established that each group of minerals have X-ray diffraction lines that are common to that group, although several lines are common to all of the clay minerals. Using this fact to an advantage, positive identification of a mineral can be accomplished when two or more lines characteristic to a mineral are detected in a diffractive pattern.

Whiteside and Marshall (52), outlining procedures for X-ray analysis of clays used the lines shown in the following chart for positive identification of specific minerals. Gruner (15) suggested the diffractive lines shown for vermiculite.

X-ray Diffraction Lines of Minerals

Common to all Minerals	Quartz	Kaolinite	Illite	Montmorillonite	Vermiculite
			angstrom units		
4.4	4.2	7.2	10.0	17.4	13.9
2.5	3.3	3.6	5.0	3.1	9.2
			3.3		5.3

The X-ray spectrometer as used by Jefferies (26) was efficient in clay mineral analysis. The diffractive pattern is measured by use of a recording goniometer and gieger tube attachment. With these attachments the angle of diffraction and intensity of the beam diffracted are measured and recorded automatically.

Cation Exchange Capacity

The cation exchange capacities of clay minerals stem directly from their structural composition. Marshall (37) found that the exchange properties of the minerals of the montmorillonite group are due to lower valency ions replacing silica in the tetrahedral sheet, leaving the mineral with a net negative charge to be satisfied by other ions.

Grim (12) states that broken bonds by mechanical loss of minerals around the edges of silica-alumina units are responsible for a portion of the exchange capacity. He states that this portion is relatively small and that the remainder and major portion of the exchange results from unsatisfied charges within the lattice structures.

The number of broken bond valences increases with a decrease in particle size. Kelley and Jenny (30) found that grinding increases exchange capacity. Johnson (27) recognizing this increase, and using graphs of curves in which one ordinate is the increasing exchange capacity and the other the decreasing equivalent spherical diameter, contends that with particles of very small diameter, such as montmorillonite, all of the exchange properties are caused by broken edge bonds.

Marshall (37) found the structure of kaolinite to have its internal charges satisfied and attributed all of its exchange properties to broken edge bonds.

The different numbers of exchange positions presented because of structural differences between the mineral groups in a given quanity of material provides a tool for identification of that material.

Ethylene Glycol Retention

Hendricks and Jefferson (19) investigated water adsorbed on external clay surfaces and between the lattice layers of clay minerals. They found, through X-ray examination, that layers of water molecules are held on the plane surfaces of clay lattices in net-like hexagonal configurations. The individual molecular layers are 3.0 Angstrom units thick and are held to the plane surfaces by a force that obeys the laws of Van Der Waal's forces.

Bradley (5) later found that certain alcohols could be introduced into a clay-water system and completely replace the water. He postulated that the alcohols assumed a layer configuration on the plane surface by CH--O bondings with oxygen atoms within the mineral.

McEwan (34) working with montmorillonite and glycerol, saturated a clay sample with an excess of ethylene glycol, boiled the mixture to remove excess water, and examined the material with X-ray equipment. He found that the long complex molecules of ethylene glycol arranged themselves parallel in a layer configuration on the surface of the lattice planes. He found them to be held by CH--O groups as postulated by Bradley (5). MacKenzie (35), working in the same laboratory as MacEwan (34), found that ethylene glycol replaces water in a molecular ratio that conforms to the hexagonal net configuration of Hendricks and Jefferson (19).

Dyal and Hendricks (10) with this background of work before them, introduced a gravimetric procedure for measuring the interlayer expansion of clays.

In their procedure, a clay saturated with an excess of ethylene glycol is subjected to evaporation and the ethylene glycol reduced to

a mono-molecular layer, covering the surface of the clay. Ethylene glycol retained by the clay is measured by weighing. The surface area of the clay is computed by insertion of the proper values into the equation.

$$\text{Surface Area} = \frac{\text{(glycol retained/gram clay)}}{\text{(area covered/gram mono-molecular layer glycol)}}$$

Bower and Gerschwend (4) later modified the procedure of Dyal and Hendricks (11) on clays, to one that is applicable to whole soils.

Electron Microscope

The revolving power of the electron microscope enables a direct magnification of 3,000 to 15,000 diameters. Further magnifications are possible by photographic means. Magnifications of this magnitude enable the eye to distinguish differences in physical characteristics of materials the size of clays.

With the electron microscope, an electron source is focused through an aperture onto a fluorescent screen. The specimen to be viewed is mounted on a thin collodion film and placed in the path of the electron beam. The specimen deflects a portion of the focused beam of electrons, and causes its image to appear as a greatly magnified shadow on the fluorescent screen.

The value of the electron microscope in identifying mixtures of materials found in soil clays was demonstrated by Bramo, Cady, Hendricks and Swendlow (6). They identified hexagonal kaolinite particles and rod shaped particles of halloysite which were indicated to be present in a sample by X-ray analysis.

Differential Thermal Analysis

When clay minerals are heated at a gradual and uniform rate, they volatilize adsorbed water at low temperatures and lattice (OH) ions at higher temperatures. The volatilization of water, either adsorbed or lattice ions, is accompanied by energy absorption. An inert material which is heated at the same rate looses water at a constant rate. A thermocouple which is inserted in two materials during heating enables a graph to be constructed showing peaks at which water is volatilized from the clays. This is indicated by the difference in the temperature of the two materials.

Endothermic peaks caused by loss of water, and exothermic peaks in the graphs caused by recrystallization within the mineral lattice, occur within definite temperature ranges that are characteristic of each mineral group. Orcel and Caillere (43) first showed these peaks to have value in clay mineral investigations. Grim, Bray and Bradley (13) have shown that the sharpness of the peaks in a mixture is dependent upon the components of the mixture. Kerr, Kulp and Hamilton (31) and Norton (41) have found that reproducible results in thermal analysis are dependent upon the packing of the material in the specimen holder.

Norton (41) in attempts to use thermal analysis as a quantitative measure of clay minerals, found that in graphs of the endothermic and exothermic peaks, the heating rate affected the height of the peaks and the temperature at which they occurred but had very little effect on the sectional area under the curve. He proposed a mathematical index to compute the quantity of specific minerals present in a mixture, using the sectional area of the curve as a criteria.

The sensitivity of thermal analysis for a given mineral is dependent upon the intensity of its thermal reaction. Orcel and Caillere (43)

examining known mixtures, found they could detect kaolinite in mixtures as low as three percent and montmorillonite in mixtures containing twenty percent montmorillonite.

Clay Minerals

The Montmorillonite Group

Ross and Hendricks (47) recognized the montmorillonite group to consist of the minerals, montmorillonite, beidellite, nontronite, saponite, and hectorite.

The montmorillonitic group has the general formula (11) of $Al_4 Si_8 O_{20} (OH)_4 (H_2O)_X$ and consists of structural units of one gibbsite sheet, $(Al_4 O_8 OH_4)$, of octahedral configuration between two silica sheets, $(Si_4 O_6)$, having tetrahedral configuration. The minerals within the montmorillonite group are characterized by substitution of other cations for those originally in either the gibbsite or the silica sheet.

Montmorillonite usually has Mg^{+2} substituted for Al^{+3} in the gibbsite sheet. Saponite is characterized by complete replacement of Al^{+3} in the gibbsite sheet with Mg^{+2}, with some replacement of Si^{+4} by Al^{+3} in the silica sheet. Hectorite is very similar to saponite, but differs because of Li^{+1} substitution for a portion of the Mg^{+2} in the gibbsite layer. Nontronite has some Al^{+3} substituted for Si^{+4} in the silica sheet, but is characterized by the substitution of Fe^{+3} for Al^{+3} in the gibbsite layer. Beidellite has the gibbsite layer intact, but has some substitution for Al^{+3} for Si^{+4} in the silica sheet.

The montmorillonite group is characterized by its swelling characteristics, caused by water adsorption (19) between the lattice layers of the clays.

The group is most easily identified by X-ray analysis. MacEwan (33) established a definite 17.4 Angstrom basal line that was detectable with as little as one percent montmorillonite. The basal exchange capacity of the montmorillonite group is higher than that of other groups of minerals. This is because of more isomorphous substitution within the lattice sheets leaving unsatisfied valence charges. Investigators (47, 8, 30) have found the exchange capacity to vary from 60 to 120 m.e. per 100 grams of clay.

Ethylene glycol retention as introduced by Dyal and Hendricks (10) has been shown by Martin (39), Martin and Russell (40), and Wilkinson and Gray (53) to be a valuable criterion in estimating specific amounts of expanding lattice colloids present.

The strong endothermic reactions measured at low temperatures by differential thermal curves are definite criteria in montmorillonite identification (31). Methods have been suggested by Norton (41) for measuring the area under the curve in the graphs of their endothermic reactions as a quantitative estimate of the mineral present.

Differentiation between minerals within the montmorillonite group can be accomplished by total chemical analysis of the constituents present (47) and use of the electron microscope.

Marshall, Shaw, Humbert and Caldwell (38), in detailed studies with the electron microscope, showed physical differences between minerals of the group. Montmorillonite, beidellite, and saponite were shown to have fluffy plates with irregular edges. Hectorite was found to have fiberous structure and nontronite, to have irregular platy structures. These observations have been confirmed by Shaw (48), Humbert and Shaw (21), and Bates, Hildebrand and Swineford (3).

The Kaolinite Group

Kerr, Kulp, and Hamilton (31) recognize four minerals in the kaolinite group: Kaolinite, dickite, nacrite and halloysite. A fifth mineral, anauxite, was considered by Ross and Kerr (46) to be a member of the group, but later investigations have caused Kerr, Kulp and Hamilton (31) to consider the mineral an impure kaolinite containing amorphous silica.

The kaolinite group has the general chemical formula of $Al_4\ O_{10}\ Si_4\ (OH)_8$. The structure (11) of the mineral group is composed of units of one gibbsite sheet, $Al_4\ O_{10}\ (OH)_8$, of octahedral configuration and one silica sheet, $Si_4\ O_6$ of tetrahedral configuration. The minerals kaolinite, dickite and nacrite differ from one another only in the manner of stacking of units in layer formations (46). The gibbsite sheet in halloysite is curled around the silica sheet, giving this mineral a tube-like structure.

Bates, Hildebrand, and Swineford (3) propose an explanation for the tube-like structures of halloysite. They state that the nonconformity of oxygen distances in the silica layer to hydroxyl-hydroxyl groups in the gibbsite sheet in kaolinite, nacrite, and dickite cause the hydroxyl groups to bend to conform to the oxygen unit spacings. In halloysite, the hydroxyl groups do not bend. These groups when extended to their full unit distances cause a curvature of the gibbsite sheet which causes the tube-like structure of halloysite.

The structural units of the kaolinite group are held very closely by chemical bonds that allow no lattice expansion by water adsorption. Although there is little isomorphous substitution of ions within the lattice units, Marshall (37) states that there is no theoretical reason

why highly reactive 1:1 lattice materials with isomorphous substitution and interlayer expansion could not exist.

Positive identification of the minerals of the kaolinite group can be obtained with either differential thermal analysis, X-ray diffraction patterns, and in the case of halloysite, the electron microscope.

With differential thermal analysis (41, 16) the loss of crystal lattice water between 575° to 700° C., causes a pronounced endothermic reaction that is characteristic to all the minerals of the kaolinite group. Orcel and Caillere (43) found this reaction to be detectable in mixtures of clay minerals where kaolinite constituted as little as three percent of the mixture.

Whiteside and Marshall (52) have identified kaolinite with X-ray diffraction patterns using the first order basal spacing of 7.2 Angstroms plus other basal lines that are characteristic to the mineral group. Bates, Hildebrand and Swineford (3) suggest identification of halloysite with X-ray diffraction patterns by 10.2 Angstrom line that collapses upon dehydration. Kelley (28) states that kaolinite cannot be detected by X-ray equipment in concentrations of less than five percent.

The low exchange capacity (3 to 12 m.e. per 100 g.) of kaolinite is rarely revealed in a mixture. Contaminations of higher exchange materials, even in minute quantities, tend to mask its low exchange properties.

The Illite Group

The name illite was proposed by Grim, Bray, and Bradley (13) in 1937 for a group of clay minerals of the mica group. It has the

general formula (16) of $K\ Al_3\ Si_3\ O_{10}\ (OH)_2\ (HOH)_X$. Illite is composed of one gibbsite sheet between two silica sheets, similar to the structure of the montmorillonitic minerals.

Grim (12) found that about one sixth of the Si^{+4} in the silica sheets is replaced by Al^{+3} from the Alumina sheet. This replacement leaves a strong negative charge within the structure of the mineral. The deficiencies within the gibbsite sheet are usually filled by K^{+1} ions.

Because of the unsatisfied charges left by the replacement of Si^{+4} by Al^{+4}, the negative charges that supplies most of the exchange positions for the mineral are found on the outer silica layers.

The presence of potassium ions between the interlayers of the mineral prevents water adsorption and expansion common to the minerals of the montmorillonite group.

The limits in range of structural and chemical characteristics of illite are thought by Grim (12) to be gradational from a well crystallized, inert mica to an expanding lattice, highly active montmorillonite.

The material selected by Grim, Bray and Bradley (13) to be a representative sample of the material they chose to call illite contains from 4.7 to 6.9 percent K_2O and has a base exchange capacity that varies from 20 to 35 m.e. per 100 grams clay. X-ray examination of the material reveals a 10.1 Angstrom lattice spacing that is unaffected by dehydration. Aldrich, Hellman, and Jackson (1) and Jackson and Hellman (22) have reported the presence of micaceous materials that, when fully hydrated, have lattice spacings of 12 to 13 angstroms. These values could be within the range of illite, or they could be evidence,

as stated by Grim, Bray, and Bradley (13), that additional separations should be made within the illite group and a new name proposed.

Vermiculite

Walker (51) states that vermiculite is a secondary mineral produced by the decomposition of mica. The structure is similar to montmorillonite, differing from it in the strength of bonding between the successive layers. Hendricks and Jefferson (18) assigned it the chemical formula of $(OH)_3$ $(Mg_{2.75}$ $Fe_{.25})$ $(Al_{1.25}$ $Si_{2.75})$ O_9 $3.5H_2O$.

Identification of the mineral by X-ray equipment is accomplished by the presence of a strong 14.1 Angstrom second order basal line that is reduced to a 10.5 Angstrom line by boiling the clay mineral in an ammonium solution (51). Walker (51) states that the maximum cation exchange capacity of the material is 130 m.e. per 100 grams of clay.

Mixed Layered Minerals

Numerous investigators (18, 14, 22, 2) have reported clay minerals with interstratified layers of more than one type clay mineral.

Grim (12) states that orientation of mixed-layered groups of minerals is possible, and that these groups could be as stable as single unit mineral groups. He contends that these mixed-layered groups may be of two types: (1) Regular repetition of layers, stacked along the C-axis, and (2) Random stratification of layers in the direction of the C-axis. Hendricks and Alexander (16) state that mixed layered structures are thought to be very common is soils.

Detection of mixed layered minerals is difficult. Careful X-ray techniques are required. Diffraction patterns of regularly interstratified layers of different minerals are a multiple of the individual

mineral units, within the composite structure (12). The pattern of randomly stratified minerals differs little from that of the dominant minerals within the stratified group.

Clay Mineral Weathering

Jackson, Tyler, Willis, Barbeau and Pennington (24) make the assumption in mineral weathering that the more resistant materials will be the last to weather and will accumulate within specific size ranges. The presence or absence of a mineral or minerals within a size range when they are present in the raw parent material indicates a stage of weathering. They also postulate that one collodial material may be parent material for a succeeding mineral, and that this type of weathering, where it occurs, is reversible.

They propose a sequence of minerals with varying resistances to weathering. The successive stages for a key to clay mineral weathering are:

1. Gypsum (also halite)
2. Calcite (also dolomite, aragonite) (apatite)*
3. Olivine-hornblende (also diopside)
4. Biotite (also glauconite, chlorite, antigorite, nontronite)
5. Albite (also anorthite, microcline, stilbite)
6. Quartz (also cristobalite)
7. Illite (also muscovite, sericite)
8. Hydrous mica-intermediates (intermediate and vermiculite)*
9. Montmorillonite (also biedellite etc.)
10. Kaolinite (also halloysite)
11. Gibbsite (also boehmite) (allophane)*

12. Hematite (also goethite, limonite)

13. Anatase (also rutile, ilmenite, corundum) (zircon)*

*Modification by Jackson, Hseung, Corey, Evans and Vander Heuvel (23).

Minerals from stages 6 to 11 of the sequence are most common in soil clays, usually with one or two minerals dominant, and with decreasing amounts of adjacent minerals with remoteness from the dominating material (24). The rate of weathering from one stage to another is considered to be a result of the factors of (temperature, water relationships, acidity, and oxidation-reduction) (the surface nature of the material).

Efforts to correlate the clay mineral group found in a soil to the factors of soil formation; climate, vegetation, relief, parent material and time, have met with varying degrees of success (20,52,55).

Humbert and Marshall (21) have found clay minerals of the same characteristics to have weathered from parent materials of widely varying chemical composition. Whiteside and Marshall (52) found similar types of minerals developed from the same parent materials but in different total amounts. Pearson and Ensminger (44) found a wide difference in species of clay minerals developed from different parent materials. Buehrer, Robertson and Deming (7) have shown the same clay mineral type to be present in several alluvial soils of widely varied origin. Grim (12) believes the effect of the parent rock on the resulting clay mineral to be relatively short lived in humid regions.

Alexander, Hendricks, and Nelson (2) examined two different parent materials in Red and Yellow, and Gray Brown podzolic soils. In each great soil group, kaolinite was found to be the dominant clay mineral with lesser amounts of other minerals present in the samples.

Winters and Simonson (55) reported the clay minerals found to be present in the subsoils of Podzols, Laterites, Chernozems and Desert soils. Contrary to Alexander, Hendricks and Nelson's (2) findings, they found the subsoils of Podzols to be dominantly illite with lesser amounts of montmorillonite and kaolinite. They found the clays in Lateritic soils to be dominantly kaolinite while those in the Chernozems were illite and montmorillonite, and those in the Desert soils were montmorillonite and illite.

Weathering of the clay minerals has been shown by Pearson and Ensminger (44), Buehrer, Robertson and Deming (7), and Humbert and Marshall (21) to be most intensive in the surface horizons of soil and to decrease in intensity with depth. This is in accord with the accepted theories of profile development and soil formation.

Particle size is thought to be a key to weathering stages. The minerals which are more resistant to weathering will persist in greater quantities in the finer sizes. The fact that the kaolinite crystals are able to grow, probably accounts for the ability of kaolinite to be more resistant to weathering than montmorillonite (24).

Pennington and Jackson (45) found a distinct separation of mineral groups into size fractions. Kaolinite was found to segregate into size fractions greater than .08 micron with most of the particles larger than .2 micron. Illite was found in all fractions but was usually segregated into fractions larger than .2 micron. Montmorillonite, on the other hand, was found to be dominant in the fractions less than .08 micron. These observations are borne out by the work of other investigators. Larson, Alloway, and Rhodes (32) found montmorillonite to dominate in clays less than .2 micron in diameter and illite in

clays 2 to .2 micron in diameter. Coleman and Jackson (9) found quartz and kaolinite to be dominant in clays 2 to .2 micron in size, with montmorillonite dominant in the less than .2 micron clays. Whiteside and Marshall (52) also noted the segregation of kaolinite into the coarser size fractions.

MATERIALS AND METHODS

Soils

The soils used in this investigation are of the Dennis and Parsons series. The two series occupy extensive areas in eastern Oklahoma. They are agriculturally prominent because their combined areas dominate the mature soils of the gently sloping to nearly level upland areas within this region.

Morphologically, the Dennis series is recognized as a southern Brunizem and the Parsons series as a Planosol in the Reddish Prairie soils. In a catena relationship, the Dennis series occupies gently sloping areas above level or nearly level slightly convex areas of Parsons. The profile description are as follows.

Dennis Silt Loam

Location: Wagoner County, Oklahoma; 3½ miles northwest of Wagoner. Six hundred feet east and two hundred fifty feet north of the southwest corner, Section 29, T18N; R18E.

Site: Gently sloping, erosional upland with a convex surface and gradient of 1½ percent. The area is a native bluestem meadow of good vigor and density.

Profile:

A_{1-1} 0-12" Very-dark-grayish-brown (10YR 3/2; 2/2 when moist) silt loam; moderate fine and medium granular; friable; porous and permeable; pH 6.0; grades to horizon below.

A_3 12-16" Very-dark-grayish-brown (10YR 3/2; 2/2 when moist) heavy silt loam; moderate fine and medium granular; friable, porous and

permeable; a few fine reddish-brown specks occur around rootlet channels and fine pores; pH 6.0; grades to horizon below.

B_1 16-26" Grayish-brown (10YR 5/2; 4/2 when moist) silty clay loam with common, medium distinct reddish-brown and strong-brown mottles; moderate, medium subangular blocky to coarse granular; friable to firm; fine black concretions and a few rounded siltstone fragments; pH 6.0; grades to horizon below.

B_2 26-31" Grayish-brown (10YR 5/2; 4/2 when moist) silty clay with many medium, distinct reddish-brown, strong-brown and yellowish-brown mottles; moderate medium subangular blocky; firm; slowly permeable; fine black concretions and a few rounded siltstone fragments; pH 6.0; grades to horizon below.

B_3 31-40" Grayish-brown (10YR 5/2; 4/2 when moist) silty clay with many distinct, coarse yellowish-brown and reddish-brown mottles: compound weak coarse blocky and moderate medium subangular blocky; firm; slowly permeable; a few fine rounded siltstone fragments; many coarse, prominent, very dark-brown accretions arranged partly in vertical pattern following the natural cracks; pH 6.0; grades to horizon below.

C_1 40-54" Grayish-brown (10YR 5/2; 4/2 when moist) clay with many coarse distinct yellowish-brown mottles, weak blocky; firm; hard when dry, plastic when wet; a few fine rounded siltstone fragments; fine concretions and medium to coarse accretions numerous; pH 6.0; grades to horizon below.

C_2 54-72" Light-brownish-gray (10YR 6/2; 5/2 when moist) light clay with many coarse, distinct yellowish-brown mottles and common, medium to coarse black concretions and accretions forming rounded pockets; weak blocky; firm; hard when dry, plastic when wet; pH 7.0. This is only partially altered shale.

C_3 72-90" Mottled brownish-yellow (10YR 6/7; 5/8 when moist) and gray (10YR 6/1; 5/1 when moist) light clay with a few medium concretions and soft, black accretions forming pockets in the mass. This is weakly blocky, partially altered shale of neutral reaction.

The parent materials are slightly acid to neutral sandy shales, sandstones, and intercalated clayey shales of Pennsylvania age. Associated with it nearby are small areas of Parsons soils on areas of nearly level relief, while the hilly lands to the north and south are occupied by Collinsville and Talihina soils. Dennis develops in materials of intermediate texture averaging about silty clay loam or light clay. Parsons forms in more clayey beds on gentle slopes and flats.

Parsons Silt Loam

Location: Mayes county, Oklahoma; 1½ miles west of Adair, 1280 feet west and 100 feet south of the northeast corner, Section 32, T23N; R19E.

Site: In nearly level erosional upland with plane to weak convex surfaces and gradient of about ½ percent. It is in a native bluestem meadow with a thick, vigorous, growth of grass.

Profile:

A_{1-1} 0-6" Dark-grayish-brown (10YR 4.5/2; 3/2 when moist) silt loam; weak to moderate medium granular; friable; pH 6.0; grades to horizon below.

A_{1-2} 6-10" Dark-grayish-brown (10YR 4/2; 3/2 when moist) silt loam; weak to moderate medium granular; friable; contains a few splotches of light-brownish gray and a few fine dark-brown specks about the root holes; pH 5.8; grades shortly to horizon below.

A_{2-1} 10-14" Light-brownish-gray (10YR 6/2; 5/2 when moist) silt loam with many medium distinct dark-brown mottles; weak medium granular; friable; permeable; pH 5.8; grades shortly to horizon below.

A_{2-2} 14-16" Light-brownish-gray (10YR 6/2; 5/2 when moist) silt loam with many medium, distinct dark-brown mottles; porous massive; friable; permeable; a few fine balls of dark-brown clay in lower one-half inch; pH 5.8; rests on horizon below.

B_{2-1} 16-22" Very-dark-grayish-brown (10YR 3.5/2; 3/2 when moist) clay with common, medium, distinct strong-brown mottles; weak coarse blocky; very compact; very slowly permeable; sides of peds strongly coated with light gray films; strong vertical cracks; pH 6.0; grades to horizon below.

B_{2-2} 22-28" Much like the layer above, but dark-brown (10YR 3.5/3; 3/3 when moist) clay with a few, very fine distinct strong brown specks and a few, fine black concretions; grades to horizon below.

B_{3-1} 28-37" Light yellowish-brown (10YR 6/4; 5/4 when moist) clay with a few, fine distinct strong-brown mottles; weak coarse blocky; very compact; very slowly permeable; a few fine rounded siltstone fragments and small black concretions; pH 6.0; grades to horizon below.

B_{3-2} 37-43" Grayish-brown (10Yr 5/2; 4/2 when moist) clay with common medium faint yellowish-brown mottles; weak coarse blocky very compact; very slowly permeable; a few fine round black concretions slightly less compact than layer above; pH 6.0; grades to horizon below.

C_1 43-66" Coarsely mottled light-gray (10YR 7/1; 6/1 when moist) strong brown (7.5 YR 5/6 when moist) and yellowish-brown (10YR 5/4; 4/4 when moist) clay; massive; firm; slowly permeable; seams of sandy clay

loam common; fine rounded siltstone chips and clusters of white gypsum crystals common; pH 6.5; grades to horizon below.

C_2 66-84" Coarsely mottled yellowish-brown (10YR 5/6; 4/6 when moist) and gray (10YR 6/1; 5/1 when moist) clay; massive; compact; slowly permeable; a few rounded chips of siltstone and small pockets of white gypsum crystals; pH 6.5. This is only partially altered clay shale but is slightly more compact than the material in the C_1 layer above.

This soil develops in soft shales or clays of Pennsylvanian age. It has formed on a very gentle slope while nearby, to the north and east are areas of Cherokee soils on the slightly concave flats with deficient surface drainage. These are lighter-colored soils with thicker A_2 horizons. To the west on less clayey material, where surfaces are convex and relief is a bit greater, Dennis soils develop.

Sample Preparation and Methods of Analysis

Clay Separation and Fractionation

Both soils used in this study were sampled in detail. Samples were taken from each horizon, allowed to air dry, and crushed to pass a 60 mesh sieve. A 400 gram representative sample of soil from each horizon was used for obtaining the clay fraction. The method used in separating the clay fraction was as follows:

A 400 gram sample selected for clay separation, was placed in an 800 ml. beaker. The sample was made into a thin paste with distilled water and titrated with 0.1 normal HCl to a pH of 3.5-4.0 (spot plate test). The sample was placed on a steam plate and treated with H_2O_2 to remove the organic material.

After frothing had ceased, the sample was left on a steam plate for two hours to remove excess H_2O_2. At the end of the period, the sample was allowed to flocculate; the filtrate very carefully decanted and the flocculated sample washed with distilled water into a Buchner funnel. Salts and excess HCl were removed by washing five to six times with distilled water. A salt free sample was indicated by a negative test for chloride with dilute $AgNO_3$. The salt free sample was then washed two times with 95% ethanol and stored in a desicator with the humidity maintained at eighty percent. The sample was stored at a high humidity to avoid caking and hardening of the sample and to aid in subsequent dispersion.

The method used for sodium saturation and dispersion was that of Jackson, Whittig and Pennington (25).

The dispersed samples were transferred to seven liter carboys and sufficient distilled water added to bring the volume in each carboy to five liters. The suspension was allowed to stand in this state for separation.

With this method of dispersion, the five liter suspension had a pH of 9.5 and needed no further treatment throughout the cycle of clay separation. Jackson, Whittig and Pennington (25) contended that boiling the sample with Na_2CO_3 for dispersion, removed small quantities of organic matter not removed by H_2O_2 treatment.

The sample was fractionated at 5 microns by sedimentation. This initial separate was further fractionated into the fractions, 5-2 micron, 2-.1 micron and less than .1 micron particles. The fractions, 2-.1 micron and less than .1 micron, were used in this investigation. The

materials 5-2 micron and larger than 5 micron were air dried and stored for future investigations.

For the initial 5 micron separation, the soil suspension in the carboy was shaken vigorously for complete dispersion and allowed to stand undisturbed for sedimentation. The material in suspension was assumed to have a specific gravity of 2.65. Tanner and Jackson's (50) nomographs were used. At a predetermined time, a siphon was lowered into the suspension to the calculated settling depth of the 5 micron particles, and the suspension containing particles less than 5 microns in diameter was siphoned off. The carboy was refilled with distilled water to the five liter mark and the separation repeated until the liquid above the settling depth of the 5 micron particles became clear at the end of the calculated settling time. It was usually necessary to make this separation six to eight times.

The siphoned material containing particles less than 5 micron in diameter was collected in eighteen liter carboys, mixed throughly, and run directly through a Sharples supercentrifuge.

The concentration of the suspension was unknown. It was therefore, necessary to run the suspension of materials through the centrifuge with it operating at a speed in excess of that necessary for .1 micron fractionation. The material in suspension could then be collected and redispersed at a known suspension. The siphoned material was run through the centrifuge at 250 ml. per minute, with the centrifuge operating at 50,000 r.p.m.. The material that passed through the machine was assumed to be less than .1 micron in diameter.

After the soil material was collected on a plastic sleeve inside the centrifuge bowl, it was removed and weighed. A sample of this

material, and sufficient other samples collected at lower operating speeds were dried to determine the moisture content of the materials collected on the sleeve at a specific operating velocity. In this manner, the clays could be weighed and redispersed into suspensions of known concentrations for further separations.

The Sharples supercentrifuge that was employed was steam driven, and after the revolutions per minute were calibrated to the steam pressure, a constant operating speed was maintained by close control of the steam pressure. A constant head of pressure for sample volume flow control was maintained to the centrifuge bowl by a vacuum siphon device from the eighteen liter carboy to the receiving flask of the centrifuge bowl.

A modification of Stoke's law (53) was used to determine the bowl speed and flow rate necessary for separation of the less than .1 micron particles from the 5 micron sedimentation separation.

Specific gravity of the less than .1 micron particles was assumed to be 2.20 (12). The Sharples supercentrifuge bowl has the following demensions:

Vertical distance of wall. . . . . 20.0 cm.

Radius of centrifuge bowl. . . . . 2.221 cm.

Radius of air column. . . . . 0.734 cm.

During the period of calibration, the suspension which flowed through the centrifuge was collected in an open flask. It was rerun through the centrifuge after calibration was complete. Care was taken in calibration to avoid particles of less than .1 micron being retained on the plastic sleeve. The proceedure was to reduce the velocity from a higher rate to a lower rate, and to reduce the flow rate from a

higher value to a lower value. Recalibration of flow rate was necessary after each cleansing of the flow rate device. Cleansing was necessary between samples to avoid contamination.

Separation of the less than .1 micron particles was made with a centrifuge bowl speed of 40,000 r.p.m.. With the insertion of the proper values into the modified Stoke's law (53), it was found necessary to pass a one percent clay suspension through the bowl at 332 ml. per minute (26° C.), to effect a .1 micron particle size fractionation.

The material of less than .1 micron which passed through the centrifuge was collected in an eighteen liter carboy and flocculated with 20 ml. of .05 N. HCl. The materials collected on the plastic sleeve were weighed, redispersed at a one percent suspension in distilled water and rerun through the centrifuge for more complete separation.

Separation of the less than .1 micron size fraction was considered complete when the weight of material retained on the plastic sleeve became constant, and the turbidity of the suspension which passed through the centrifuge bowl was reduced to clarity. It was usually necessary to pass the resuspended material through the centrifuge six to eight times to attain these conditions. Whitt and Baver (53) found this number of separations satisfactory under similar conditions.

The supernatant liquid was carefully decanted from the flocculated clay of less than .1 micron particle size. The flocculated clay was then placed in 100 ml. centrifuge tubes and centrifuged to further reduce the volume. The supernatant liquid was decanted and the clay transferred to 2000 ml. Erlenmeyer flasks for storage until samples were taken for examination.

To separate the 2-.1 micron fraction, the 5-.1 micron fraction was redispersed, placed in a seven liter carboy, and separated by sedimentation into 5-2 micron and 2 +.1 micron fractions. The separation was made in the manner used for the initial 5 micron separation.

After separation, the 2-.1 micron-clay suspension was flocculated with 5 ml. portions of .05 N HCl, reduced in volume by centrifuging, and stored in Erlenmeyer flasks. The 5-2 micron fractions were flocculated, air dried, and transferred to small envelopes for storage.

Preparation of Samples for X-ray Analysis

In preparing the samples for X-ray analysis, iron oxides were removed from 0.5 gram equivalent volumes of the flocculated sample stocks obtained in the initial clay separations. For iron oxide removal, each sample was dispersed with 50 ml. of distilled water in a 100 ml. centrifuge tube. Two grams of sodium hydrosulfite were added to the suspension, and the centrifuge tube was placed in a water bath, with the temperature maintained at 40-45° C.. The suspension in the centrifuge tube was stirred frequently over a thirty to fifty minute period, or until the clay was bleached.

After iron oxide removal, the sample was treated with dilute HCl to remove interfering cations. The bleached sample was shaken ten to fifteen minutes with 50 ml. of .02 N. HCl. It was again centrifuged, the excess HCl decanted, and then washed free of all excess salts by dispersing in 25-50 ml. portions of distilled water, centrifuging and decanting the wash water. The sample was considered free of salts by a negative test for chlorides with $AgNO_3$.

The clays were then saturated with NH_4 ions for chlorite-vermiculite differentiation and Ca ions and glycerol for expanding and non-expanding

lattice determinations. For cation saturation, a 100 to 150 mg. sample of the iron oxide free clay was boiled gently for fifteen minutes with a fifty ml. portion of 1.0 N NH_4Cl or an equal volume of 1.0 N $CaCl_2$. To this clay-$CaCl_2$ mixture was added four to six drops of glycerol for lattice expansion. After boiling, the sample was allowed to cool, centrifuged and the supernatant liquid was decanted.

An oriented clay mount was prepared by suspending the cation saturated clay in two to three ml. of distilled water, pipetting a sample of it onto a prepared microscope slide, and allowing the sample to air dry on the slide, inside a dust free enclosure.

The microscope slide was prepared by cleansing with acetone, wiping dry with a lintless towel, and providing a 1 inch by 1 inch boundry to contain the sample with masking tape.

Preparation of Samples for Differential Thermal Analysis

A one gram equivalent volume of stock clay sample was Ca ion saturated by boiling gently for fifteen minutes with 75 ml of 1.0 N $CaCl_2$. The sample was then centrifuged, the supernatant liquid decanted, and the clay was washed with distilled water to remove the excess salts. The salt free clay was dried at low temperatures (less than 80° C.) crushed to pass a 60 mesh sieve, and placed in a stoppered, soft glass test tube.

The samples contained in the stoppered, soft glass test tubes and the oriented clays on the microscope slides were sent to Dr. S. B. McCaleb at the North Carolina State Experiment Station for X-ray spectrographs and differential thermal analysis.

Preparation for Electron Microscope Examination

The procedure of clay preparation for viewing in the electron microscope is essentially that of MacKie, Chatterjee and Jackson (36).

A sample of stock clay was treated with .02 N HCl to saturate the exchange complex with H^+ ions. It was then washed free of excess salts with small portions of distilled water. The sample was dispersed by adding saturated NaOH dropwise until a pH of 9.5 was reached (Spot plate test). The clay material was then diluted to .01 percent suspension and was ready for specimen mounting.

For mounting, a drop of the clay suspension was allowed to dry over night on a film of collodian supported by a 200 mesh screen.

The screen is prepared by adding a 10 percent solution of collodian in amyl acetate, dropwise, to a water surface and allowing a film to form. A small section of screen, precut to fit the receptacle of the electron microscope, is then dropped onto the film and picked up on a microscope slide in a manner that leaves the film covering the surface of the screen and the slide.

The screen containing the air dried specimen is fitted into a receptacle and inserted directly into the electron microscope. The electron microscope used was a R.C.A. Console model type E.M.C.-2 operating at 30,000 volts. It is capable of magnifications of approximately 5,000 diameters.

Photographs of the specimens were made by direct exposure of photographic film to the electron field.

Chemical Analysis

Base exchange capacity of the two size fractions was determined by the barium replacement method of Whitt and Baver (53).

A one gram sample was saturated with hydrogen; the hydrogen ions replaced with barium from a N.$BaCl_2$ solution, and the acidity of the filtrate titrated with 0.1 N. NaOH. Replacement with barium was continued until the NaOH required to titrate the replaced H^+ ions became constant.

The sum of the total me. of hydrogen replaced divided by the sample weight is equal to the me. of exchange per gram soil.

The method of Dyal and Hendricks (10) was used to determine clay surface areas. Using their method, total surface areas and internal surface areas of the clay materials were determined.

The procedure consists of drying two samples of a hydrogen saturated clay in such a manner that the mineral structure of one sample is collapsed, and the structure of the other sample remains intact. These two samples are saturated with ethylene glycol and allowed to reach equilibrium. The excess glycol is evaporated from the clays in a vacuum system. Evaporation of the excess, leaves a monomolecular layer of glycol covering the surface areas of the clays. The weight of ethylene glycol retained (mg. glycol per gram clay) is a measure of the total surface area. The weight of glycol retained (mg. glycol per gram clay) by the sample with the structure intact minus the weight of glycol retained (mg. glycol per gram clay) by the sample with collapsed structure is a measure of interlayer surface area, i.e., interlayer expansion.

The method used for determination of non-exchangeable potassium is a modification of the procedure of Hall (15).

A .5 gram hydrogen saturated sample was thoroughly mixed with .5 gram of NH_4Cl and 1.5 grams of $CaCo_3$, using an agate mortar and pestle.

The mixture was washed into a platinum crucible with an additional .5 gram of $CaCO_3$.

The crucible and its contents were placed in an electric furnace and the temperature increased slowly until NH_3 fumes ceased to be evolved. The temperature of the furnace was then raised to 800° C. and maintained at this temperature for 60 minutes. At this temperature $CaCl_2$ formed in the reaction, $NH_4Cl + CaCO_3 \longrightarrow CaCl_2$, fuses with the mass of the sample.

The crucible was removed from the furnace and allowed to cool. The fused cake was slaked with hot distilled water and transferred to a porcelain dish. The cake was then thoroughly crushed with an agate pestle, and the sample was washed five times with hot distilled water by decantation. The slaked material was transferred to a filter and further washed with hot water. The filtrate was added to the decanted solution. Four hundred milliliters of total wash water were sufficient for the removal of the fused alkalies.

A 600 ml. beaker containing the filtrate was placed on a steam plate and warmed. A sufficient volume of concentrated $(NH_4)2CO_3$ solution was added to precipitate the calcium in the solution. The contents of the beaker was then filtered through number two Whatman filter paper into an 800 ml. beaker. The filtrate was washed free of chlorides with hot distilled water.

The 800 ml. beaker was placed on a hot plate, and its contents evaporated to less than 100 ml. in volume. The solution was then transferred into a 100 ml. volumetric flask and brought to volume with distilled water. This solution was run directly through a Perkin-Elmer flame photometer, and the potassium content determined.

Mechanical Analysis

The mechanical analysis was run by the pipette method of Olmstead, Alexander and Middleton (42). To determine the less than .1 micron fraction the centrifuge method of Steele and Bradford (49), was used.

Specific gravity of the .1 micron particles was assumed to be 2.20. Time of centrifuging was measured from the time the centrifuge was turned on until it was turned off. No interpolations were made for the periods of acceleration and deceleration.

At the end of the calculated centrifuging time, a calibrated 5 ml pipette was lowered to the depth of the sedimented .1 micron particles and slowly filled (15 seconds) by gentle suction. The contents of the pipette were emptied into a weighed evaporating dish. The pipette was washed twice with distilled water and the washings added to the evaporating dish. The dish and its contents were dried at 105° C. and weighed. The percent clay was determined by the following formula:

$$\frac{(V)\ (C-D)}{(X)}\ \frac{(100)}{(S)} = \text{Percent clay}$$

V - Volume of suspension in ml.

C - Weight clay in dish in grams

D - Weight of dispersing agent in grams

X - Volume of the pipette in ml.

S - Oven dry sample weight in grams.

RESULTS AND DISCUSSION

Particle Size Distribution

The particle size distribution of the Dennis profile is given in Tables I and II.

Table I. Particle Size Distribution*, Dennis Silt Loam

		Size Class and Diameter of Particles (in mm.)					
Depth	Field Horizon Designation	Coarse and Med. Sand 2-.25	Fine Sand .25-.1	Very Fine Sand .1-.05	Coarse Silt .05-.02	Fine Silt .02-.002	Clay < .002
inches		%	%	%	%	%	%
0-12	A_1	5.4	6.3	12.7	37.0	18.8	19.8
12-16	A_3	5.0	4.6	11.3	36.8	18.8	23.5
16-26	B_1	6.1	3.8	9.6	30.0	19.1	31.4
26-31	B_2	2.8	2.5	6.9	24.6	20.3	42.9
31-40	B_3	3.8	2.6	6.5	23.8	21.2	42.1
40-54	C_1	4.0	3.2	6.8	23.5	19.4	43.1
54-72	C_2	4.4	2.8	6.4	25.2	19.8	41.4
72-90+	C_3	6.4	3.2	6.9	25.9	21.0	36.6

*Mechanical analysis by S. C. S. laboratory, Beltsville, Maryland. Samples S530K-73-20-(1-8).

These data show that the clay sized fraction was more active in the soil developing process than the other particle size. The fine silt sized fraction shows a loss of materials in the A_1 and A_3, the horizons of active weathering.

The gain in percent coarse silt shown in the A_1, A_3 and B_1 horizons is a reflection of the loss of clay sized materials from these horizons by illuviation.

The changes that have occurred in the clay sized particles are further broken down in Table II. These data show that a high percentage of the clay in each horizon, except in the slightly weathered parent

material, consists of particles less than .1 micron in diameter. Illuviation that was evident in the total clay fraction in Table I is shown in Table II to be confined to the fine clays.

Table II. Particle Size Distribution, Clay Fraction, Dennis Profile

Horizon	Depth	Total Clay 2.0 Micron	Fine Clay** <.1 Micron	Coarse Clay 2-.1 Micron	Fine Clay* <.1 Micron	Ratio Fine Clay / Coarse Clay
	inches	%	%	%	%	
A_1	0-12	19.8	15.3	4.5	77.2	3.4
A_3	12-16	23.5	15.0	8.5	63.8	1.8
B_1	16-26	31.4	23.6	7.8	75.1	3.0
B_2	26-31	42.9	30.2	12.7	70.2	2.4
B_3	31-40	42.1	26.6	15.5	63.1	1.7
C_1	40-54	43.1	25.4	17.7	56.6	1.4
C_2	54-72	41.4	18.8	22.6	45.6	.8
C_3	72-90+	36.6	13.4	23.2	36.6	.6

*% of total clay fraction.
**% of total soil

The coarse clays in the Dennis profile exhibit a consistent and continuous decrease from the slightly weathered parent material to the topsoil. This decrease, which is very pronounced, is attributed to weathering. Other data, to be discussed later, reveal some interesting features about this decrease in coarse materials.

The particle size distribution of the Parsons profile is given in Tables III and IV. The data for the Parsons profile show several features that were also exhibited in the Dennis profile.

Clay eluviation and accumulation is very pronounced in the Parsons profile. The clay accumulation in the Parsons profile is reflected in all the size separates of the profile. All size separates, other than clay, show decreases in percentage content in the B_{2-1} and B_{2-2} horizons where maximum clay accumulation has occurred.

The clay fraction in each horizon of the Dennis profile (Table II) was shown to contain a high proportion of particles less than .1 micron

Table III. Particle Size Distribution*, Parsons Silt Loam

Depth	Field Horizon Designation	Size Class and Diameter of Particles (in mm.) Coarse and Med. Sand 2-.25	Fine Sand .25-.1	Very Fine Sand .1-.05	Coarse Silt .05-.02	Fine Silt .02-.002	Clay < .002
inches		%	%	%	%	%	%
0-6	A_{1-1}	2.1	8.5	6.8	38.6	31.8	12.2
6-10	A_{1-2}	3.2	8.6	6.6	36.8	32.4	12.4
10-14	A_{2-1}	5.5	7.8	6.2	35.5	31.1	13.9
14-16	A_{2-2}	7.2	6.7	5.0	28.5	27.0	25.6
16-22	B_{2-1}	0.8	3.6	2.8	17.0	18.0	57.8
22-28	B_{2-2}	0.6	3.4	2.6	15.6	18.5	59.3
28-37	B_{3-1}	1.1	5.3	4.0	20.8	19.4	49.4
37-43	B_{3-2}	2.5	7.0	5.3	25.8	20.9	38.5
43-66	C_1	4.3	7.8	6.1	26.3	19.2	36.3
66-84+	C_2	2.2	8.0	6.2	25.2	18.1	40.3

*Mechanical analysis by S.C.S. laboratory, Beltsville, Maryland. Samples S530K-49-2-(1-10).

in diameter. The Parsons profile (Table IV) exhibits a similar feature. A notable difference between the two profiles is the proportion of the clay fraction of the Parsons parent material that is made up of particles less than .1 micron in diameter. The ratio of fine clay to coarse clay in the slightly weathered residuum of the Parsons is much higher than that of the Dennis. The Dennis slightly weathered residuum contains 36.6% fine clay as compared to 89.1% in the Parsons profile. After the clay has weathered through the initial stages of profile development, horizons C_3, C_2 and C_1 in the Dennis profile, the percentages of fine clay content in the other horizons is more nearly equal the clay content of corresponding horizons in the Parsons profile.

When the processes of eluviation and illuviation in the Dennis profile were confined to particles of < .1 micron in diameter. The

date in Table IV shows that these processes have occurred in both of the size separates of the clay fraction in the Parsons profile

Table IV. Particle Size Distribution, Clay Fraction, Parsons Profile

Horizon	Depth	Total Clay 2.0 Micron	Fine Clay** < .1 Micron	Coarse Clay 2-.1 Micron	Fine Clay* < .1 Micron	Ratio Fine Clay / Coarse Clay
	inches	%	%	%	%	
A_{1-1}	0-6	12.2	10.8	1.4	88.5	7.7
A_{1-2}	6-10	12.4	10.9	1.5	87.8	7.2
A_{2-1}	10-14	13.9	7.2	6.7	51.7	1.1
A_{2-2}	14-16	25.6	22.4	3.2	87.5	7.0
B_{2-1}	16-22	57.8	44.4	13.4	76.8	3.3
B_{2-2}	22-28	59.3	46.1	13.2	77.8	3.5
B_{3-1}	28-37	49.4	41.8	7.6	84.8	5.6
B_{3-2}	37-43	38.5	34.3	4.2	89.1	8.1
C_1	43-66	36.3	32.2	4.0	89.0	8.1
C_2	66-84+	40.3	35.9	4.4	89.1	8.1

* % of total clay fraction.
** % of total soil

The ratio of fine clay to coarse clay in the Parsons profile shows that not only has illuviation occurred in the coarse clays, but it has proceeded at a rate in excess of illuviation of the fine clay in the B_{2-1} and B_{2-2} horizons. The coarse clays of the Parsons have a low ratio of coarse clay to fine clay in the A_{2-1} horizon. This horizon of intensive eluviation of fine clay, has an accumulation of coarse clay. A similar feature is exhibited in the Dennis profile, although it is not as pronounced.

Chemical Measurements

The results of some chemical measurements in the Dennis profile are given in Table V. The organic carbon content of the Dennis profile is high in the A_1 and A_3 horizons and gradually decreases with increasing depth.

Table V. Chemical Measurements* of the Dennis Silt Loam

Depth	Field Horizon Designation	pH	Organic Carbon	Exchangeable cations (me. per 100 gm. soil) Ca	Mg	K	Na	H	Sum	Base Saturation
inches			%							%
0-12	A_1	5.4	1.48	7.3	2.7	0.2	0.2	7.8	18.2	57
12-16	A_3	5.4	1.13	7.5	2.8	0.2	0.4	7.6	18.5	59
16-26	B_1	5.6	0.74	9.0	3.1	0.3	0.6	7.6	20.6	63
26-31	B_2	5.8	0.49	11.4	4.6	0.3	1.0	7.9	25.2	69
31-40	B_3	5.9	0.31	12.4	5.0	0.3	1.1	7.1	25.9	72
40-54	C_1	6.3	0.18	14.5	5.8	0.4	1.4	6.0	28.1	79
54-72	C_2	6.6	0.17	14.6	6.6	0.3	1.4	4.5	27.4	84
72-90+	C_3	6.6	0.07	12.9	6.2	0.3	1.3	3.7	24.4	85

*Analysis by the S.C.S. laboratory, Beltsville, Maryland. Samples S53-OK-73-20-(1-8).

Table VI. Chemical Measurements* of the Parsons Silt Loam

Depth	Field Horizon Designation	pH	Organic Carbon	Exchangeable cations (me. per 100 gm. soil) Ca	Mg	K	Na	H	Sum	Base Saturation
inches			%							%
0-6	A_{1-1}	5.7	1.15	5.8	0.8	0.1	0.1	6.3	13.0	52
6-10	A_{1-2}	5.2	0.63	2.9	0.8	0.1	0.4	7.0	11.2	38
10-14	A_{2-1}	5.5	0.38	2.4	0.8	0.2	0.5	6.2	10.1	39
14-16	A_{2-2}	5.6	0.50	4.8	2.3	0.3	1.3	8.6	17.3	50
16-22	B_{2-1}	5.4	0.87	12.5	5.9	0.5	3.4	16.2	38.5	58
22-28	B_{2-2}	5.3	0.73	14.1	6.8	0.4	3.8	12.2	37.3	67
28-37	B_{3-1}	5.3	0.32	12.0	6.2	0.4	3.3	9.6	31.5	70
37-43	B_{3-2}	5.4	0.17	9.6	4.8	0.4	2.6	5.2	22.6	77
43-66	C_1	5.4	0.11	9.7	4.8	0.4	2.3	6.2	23.4	74
66-84+	C_2	5.6	0.06	12.6	6.1	0.4	2.5	5.8	27.4	79

*Analysis by the S.C.S. laboratory, Beltsville, Maryland. Sample S53-OK-49-2-(1-10).

As weathering and profile development has proceeded, base saturation has decreased from 85% in the slightly weathered parent material to 57% in the topsoil. The decrease in base saturation was accompanied by a change in pH. The profile became more acid as basic elements were lost.

These data show calcium to be the dominant basic exchangeable cation present on the clays. The amounts of all basic cations in the soil except potassium have declined as the soil developed. The loss of basic cations in the soil is reflected in a gain in hydrogen ions present on the clays.

Data on the chemical measurements of the Parsons profile are presented in Table VI. The data given on the Parsons profile are similar to those presented for the Dennis. There are striking variations between the profiles which show the Parsons to be more highly weathered and subsequently more strongly developed morphologically. The principal variation between the two profiles is the increase in percent organic carbon and each exchangeable cation in the Parsons profile that has accompanied the illuvial processes in the development of the B_{2-1} and B_{2-2} horizons. The exchangeable magnesium ions appear to have been particularly affected.

The parent material of the Parsons is more acid than that of the Dennis. The parent material of the Parsons has a pH value of 5.6 as compared to 6.6 in the Dennis. The Parsons profile exhibits a more acid nature in each horizon than does the Dennis, except in the A_{1-1} horizon which is less acid than the A_1 in the Dennis profile. The percent base saturation of the slightly weathered parent material of

the Parsons profile is lower than that of the Dennis, 79% as compared to 85%. Base saturation in the Parsons profile decreases to 38% in the A_{1-2} horizon. This is a reflection of the intensive eluviation that has occurred.

Cation Exchange Capacity

The sum of the exchangeable cations in the horizons of both profiles reflects the number of exchange positions present in that horizon. These exchange positions may be present in a small quantity of a highly reactive material or as a large quantity of a material that exhibits few exchange positions.

The amount of clay material present in each size separate of the two profiles was presented in Tables II and IV. The sum of their combined exchange positions was presented in Tables V and VI. In Table VII, the exchange capacity of each clay sized separate of each horizon of the two profiles is presented.

Table VII. Cation Exchange Capacity of the Two Clay Fractions, Dennis and Parsons Profiles

Dennis			Parsons		
Field Horizon Designation	Coarse Clay 2-.1 Micron	Fine Clay < .1 Micron	Field Horizon Designation	Coarse Clay 2-.1 Micron	Fine Clay < .1 Micron
	me. per 100 gm. clay			me. per 100 gm. clay	
A_1	14.7	53.2	A_{1-1}	13.2	53.7
A_3	13.9	51.4	A_{1-2}	15.1	51.5
B_1	16.2	52.1	A_{2-1}	11.0	51.2
B_2	16.7	51.5	A_{2-2}	15.7	53.4
B_3	17.1	51.4	B_{2-1}	23.4	52.4
C_1	17.6	53.1	B_{2-2}	18.9	51.7
C_2	19.6	55.7	B_{3-1}	20.8	55.3
C_3	15.4	53.1	B_{3-2}	16.7	52.3
Mont.*	68.0	75.4	C_1	23.0	55.0
Illite*	22.2	27.5	C_2	16.8	52.7

*Reference Clay Minerals

A striking feature in the exchange capacities of the two clay separates of the two profiles is the similarity of the exchange capacities of the fine clays. In the A_1 horizons of both profiles the exchange capacities of the fine clays are very similar. There is little variation between horizons in the exchange capacity of the fine clays in either profile.

The coarse clays in both profiles exhibit less exchange than the fine clays. There is more variation in exchange capacities in the coarse clays between horizons than there is in the fine clays. The coarse clays of the slightly weathered parent material of each profile have low exchange values. The horizons of initial weathering above this material, in each case have increased exchange capacities.

The horizons of illuviation in both profiles have increased exchange capacities. In Table II, the coarse clays of the Dennis profile were shown to have been intensively weathered into smaller sized particles in the horizons of illuviation. This weathering may have also included weathering of clay minerals with low exchange reactivities into minerals of the same particle size grouping that exhibit higher exchange reactivities.

The coarse clays of the Parsons profile, Table 4, were shown to have undergone eluviation. The increases in exchange capacities of the B horizons of this profile may be the effect of illuviation of materials that have a higher exchange reactivity. The increases shown in the horizons of both profiles are similar, an increase in the upper C horizon and in the illuvial B. At least one of the two mentioned processes, illuviation or mineral weathering, has occured in each profile.

Whether both of the processes have or have not occurred concurrently in each profile must be drawn from other data.

The exchange capacities of two reference clay minerals, montmorillonite and illite are shown in Table VII. The exchange capacity of kaolinite has been found by Grim (12) to be from 3 to 12 me. per 100 grams.

From the exchange capacity values of the reference clays given, it is evident that the fine clays are a mixture of high and low exchange capacity minerals. The coarse clays are composed of clay minerals that have low exchange values with varying amounts of a higher exchange material. Other determinations are necessary to estimate relative amounts of each mineral group present.

Non-exchangeable Potassium

Non-exchangeable potassium has been thought to be a key to the illite and micaceous clay mineral content of soils. Grim, Bray and Bradley (13) have found illite to vary from 4.7 to 6.9 percent K_2O. An arbitrary value of 6% K_2O has been used by Grim (12) and by Wilkinson and Gray (53) in quantitative estimations of amounts of this mineral present in mixtures. Micaceous materials present in soils may contain higher percentages of K_2O within their mineral structure.

The non-exchangeable potassium contents of the two clay fractions of the Dennis and Parsons profiles are presented in Table VIII.

The non-exchangeable potassium content is very similar in the coarse clays of both profiles. A feature in the K_2O content of the coarse clays of both profiles is the .3 percent decrease shown in the A_3 of the Dennis and the A_{1-2} of the Parsons. This horizon of the Dennis profile also has decreased exchange capacity as shown in Table VII.

The percent K_2O content of the fine clays is much lower than that of the coarse clays in both profiles. The K_2O content of the fine clays in the Dennis, however, is higher than the K_2O content of the Parsons.

Table VIII. Non-exchangeable Potassium Percentage Found in the Two Clay Fractions of the Dennis and Parsons Profiles

Dennis			Parsons		
Field Horizon Designation	Coarse Clay 2-.1 Micron	Fine Clay <.1 Micron	Field Horizon Designation	Coarse Clay 2-.1 Micron	Fine Clay <.1 Micron
	%	%		%	%
A_1	4.6	2.4	A_{1-1}	4.2	1.6
A_3	4.3	1.9	A_{1-2}	3.9	1.8
B_1	4.6	2.0	A_{2-1}	4.3	1.7
B_2	4.6	1.8	A_{2-2}	4.0	1.6
B_3	4.6	2.0	B_{2-1}	4.2	1.6
C_1	4.5	2.4	B_{2-2}	4.2	1.6
C_2	4.6	2.4	B_{3-1}	4.1	1.6
C_3	5.0	1.7	B_{3-2}	4.0	1.8
Mont.*	0.0	0.0	C_1	3.8	1.7
Illite*	7.5	6.8	C_2	2.6	3.1

*Reference minerals

The K_2O content of the two clay fractions in the slightly weathered parent material of both profiles is interesting. The more acid Parsons has a high percentage initially in the fine clays with a lesser amount in the coarse clays while the reverse of this is true in the Dennis profile. The major changes that occur in the K_2O content of each separate of the clays in both profiles occur in the horizons of the initial weathering of the slightly altered parent materials.

Ethylene Glycol Retention

Of the methods used for clay mineral identification and estimation, ethylene glycol retention revealed more variation between horizons than

any other analytical procedure. The weathering of larger particles into smaller particles and the presence of expanding lattice structures presents a larger total surface area that is measurable by the ethylene glycol retention process.

The data on surface area values were derived by insertion of values from analytical results into the following formulas of Dyal and Hendricks (10).

1. Total surface area - Square meters per gram sample =

$$\frac{\text{Weight ethylene glycol retained unheated sample (gm.)}}{\text{Weight vacuum dried unheated sample (gm.)} \times .00031}$$

2. External surface area - Square meters per gram sample =

$$\frac{\text{Weight ethylene glycol retained heated sample (gm.)}}{\text{Weight vacuum dried unheated sample (gm.)} \times .00031}$$

3. Internal surface area = (Total surface area) - (External surface area).

Data acquired by the ethylene glycol retention process on the surface areas of the two clay separates of the Dennis profile are presented in Table IX.

Table IX. Surface Areas of the Two Clay Fractions as Measured by Ethylene Glycol Retention, Dennis Profile

Field Horizon Designation	Coarse Clay $(m)^2$ per gm.		Fine Clay $(m)^2$ per gm.	
	Total	Internal	Total	Internal
A_1	61	30	258	157
A_3	69	42	237	145
B_1	106	64	252	140
B_2	75	34	241	168
B_3	86	59	245	160
C_1	92	41	333	201
C_2	83	37	376	252
C_3	69	33	357	219
Mont.*	502	408	557	504
Illite*	176	77	215	38

* Reference Minerals

The total surface areas of the two clay sized fractions of the Dennis substantiate weathering of clay sized particles in the processes of soil development.

The total surface area of the fine clay is reduced from 357 $(m)^2$ per gram in the C_3 horizon to 237 $(m)^2$ per gram in the A_3 horizon. This decrease in total surface area is very significant. It agrees, by horizon, with the loss of coarse clay sized particles that was shown in Table II.

The eluviation of the fine clays, shown to be apparent in Table II is not detectable in ethylene glycol retention values of that fraction. Eluviation apparently has occurred as an indiscriminate physical displacement of all particles within the range of the fine clay. However, the drop in total surface area between the C_1 and B_3 horizons may be evidence that the coarser sized particles within the fine clays were those which were illuviated.

Total surface area data on the coarse clays suggest weathering losses of the finer sized particles from this fraction. In the C_3 horizon of the slightly weathered parent material, the particle sizes are very coarse. With the initial weathering that has occurred in the C_2 and C_1 horizons there has been an increase of smaller sized particles within the coarse clay fraction. The data shows that from the B_3 horizon to the surface, smaller sized particles of the coarse clay fraction were lost to the fine clay sized separate. The larger clay sized particles left within the coarse clays exhibit decreasing values of total surface area.

The evidence mentioned does not fully explain the increase in total surface area in the B_1 horizon in the coarse clays. Illuviation

of coarse clay particles was not evident in the data of Table II. The B_1 horizon contains less coarse clay than the A_3 horizon (Table III). Illuviation of finer sized particles into this horizon is therefore eliminated as an explanation. This increase in total surface area is noted in the coarse clays of the Parsons profile, Table X, at the same soil depth.

The data on the Parsons profile suggests that illuviation of smaller sized particles into this soil depth may have occurred. Illuviation, however, does not explain the increase in total surface area in the A_{2-2} horizon, (Table X). Examination of the profile descriptions of the two soils at the depths in question shows both of them to be mottled and to have black concretions present. The presence of these mottles and ferruginous concretions suggests that this depth is moist much of the year, enabling intensive physio-chemical reactions to occur. The data suggest intensive weathering of the coarse clays at this soil depth.

Table X. Surface Areas of the Two Clay Fractions of the Parsons Profile as Measured by Ethylene Glycol Retention

Field Horizon Designation	Coarse Clay $(m)^2$ per gm.		Fine Clay $(m)^2$ per gm.	
	Total	Internal	Total	Internal
A_{1-1}	49	21	404	281
A_{1-2}	68	28	418	292
A_{2-1}	78	46	367	244
A_{2-2}	102	71	368	240
B_{2-1}	151	14	367	213
B_{2-3}	78	47	384	253
B_{3-1}	106	50	383	263
B_{3-2}	63	11	373	271
C_1	108	36	384	278
C_2	85	23	344	221

The total surface area values of the fine clays of the Parsons profile show a decrease in particle size from the slightly weathered

parent material to the surface.

The total surface area per gram clay of the fine clays of both profiles is similar in the slightly weathered parent materials that each has developed from. The surface areas of the fine clays of the Parsons show that there has been weathering of this size material in the processes of soil development.

The increases in total surface areas shown in the fine clays may be the result of weathering fine clay sized materials into smaller sized particles, or it may be the result of weathering of non-expanding lattice structures into expanding lattice type structures with their additional internal surfaces.

In the A_{2-1}, A_{2-2} and B_{2-1} horizons, the horizons that were suggested to have had extensive weathering of the coarse clay materials, the fine clays show an increase in particle size. The increase in particle size is shown by the decrease in total surface area per gram clay. The data suggest that coarse materials weathered into particles within the size range of the fine clay separate within these horizons.

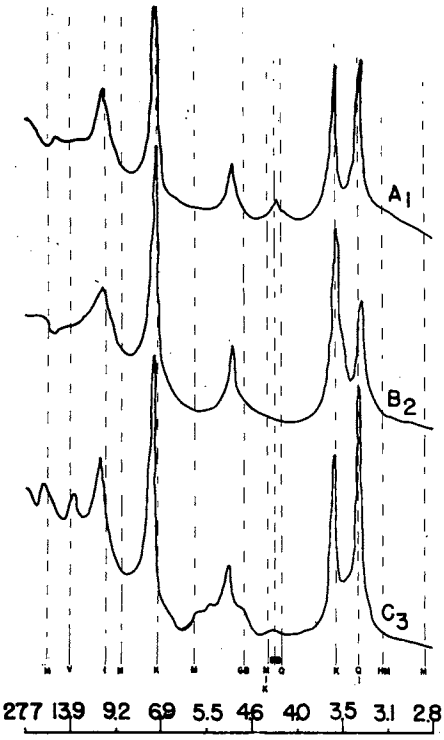
The data on internal surface areas as shown in Tables IX and X indicates, to a degree, the interlayer surfaces of expanding lattice material. An interesting feature is the similarity of internal surface area presented by the fine clays of the slightly weathered parent material of both profiles. The fine clays of Dennis have 219 $(m)^2$ per gram internal surface area while those of Parsons have 221 $(m)^2$ per gram internal surface area. The similarity of these two values is short lived in the processes of weathering and profile development that has occurred in these two profiles.

The data presented thus far has shown measurements of the chemical and physical characteristics of the clay sized materials examined. The proceedures used measure the sum of the characteristics of all of the minerals present. No analysis has been performed to measure the characteristic of a specific mineral. X-ray spectrographs and differential thermal analyses were used in this study for the determination of the presence of specific minerals in the two clay separates of each horizon of the two profiles.

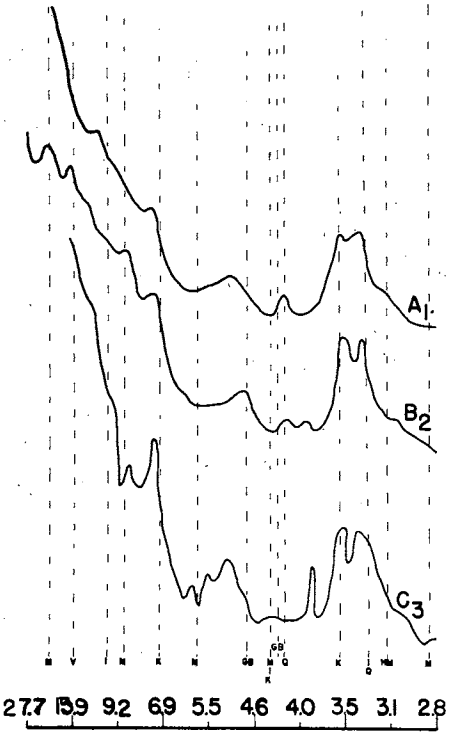
X-ray spectrographs indicated the presence of minerals not detectable by other methods of analysis. This is true of quartz, vermiculite, halloysite, and hydrous micas.

X-ray and Differential Thermal Analysis

Modified tracings of some of the X-ray spectrographs are shown in Figures 1-4. The tracings shown in Figure 1, Dennis coarse clay are typical of the spectrographic patterns.



Angstroms
Fig. 1. Dennis coarse clay
X-ray Spectrograph



Angstroms
Fig. 2. Dennis fine clay
X-ray Spectrograph

There are no abrupt changes either in peaks or intensity of diffraction through the depth of the profiles. Differences in intensity of diffraction as reflected by the height of the peaks, change gradually from one horizon to the next.

Identification of a mineral is accomplished by the occurrance of a peak at an angstrom unit spacing that is characteristic to that specific mineral. The A_{1-1} horizon of the Parsons profile has a very diffuse pattern that indicates intensive weathering.

The data presented in Tables XI and XII are the result of X-ray and differential thermal examinations for the specific mineral present in the two size fractions of each horizon of the Dennis and Parsons profiles.

Table XI. Clay Minerals* Found to be Present in the Two Clay Fractions of the Dennis Profile by X-ray and Differential Thermal Analysis**

Field Horizon Designation	Coarse Clay		Fine Clay	
	X-ray	Differential Thermal	X-ray	Differential Thermal
A_1	K(I Q M)	K	M(K M/I)	M (K)
A_3	K(I V M Q)	K	M(V H Q)	M (K)
B_1	K(I Q)	K	M(V Hm H)	M K
B_2	K(I Q M)	K	M(V K)	M K
B_3	K(I V Q)	K	M(I K)	M K
C_1	K(I M)	K	M(I K)	M K
C_2	K(I M)	K	M(H K)	M K
C_3	K(I Q M)	K	M(H K)	M K

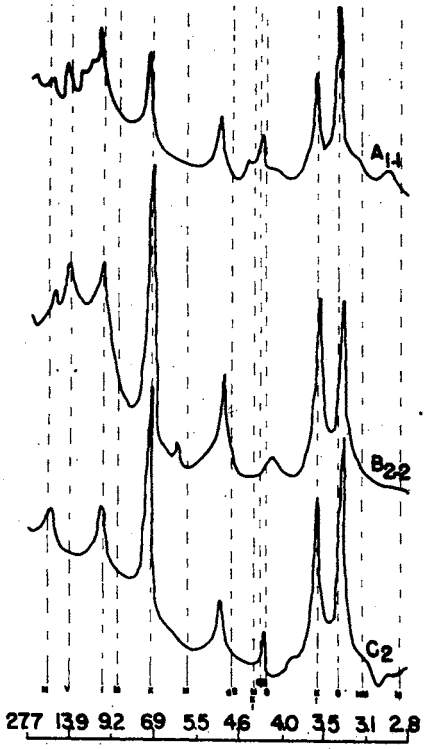
*M-Montmorillonite, K-Kaolinite, I-Illite, V-Vermiculite, Q-Quartz, Hm-Hydrous Micas, H-Halloysite, M/I-Mixed-layered Minerals. Dominant minerals present, outside parenthesis, other minerals present are enclosed by parenthesis.

**Interpretation of data by Dr. S. B. McCaleb. North Carolina State Experiment Station.

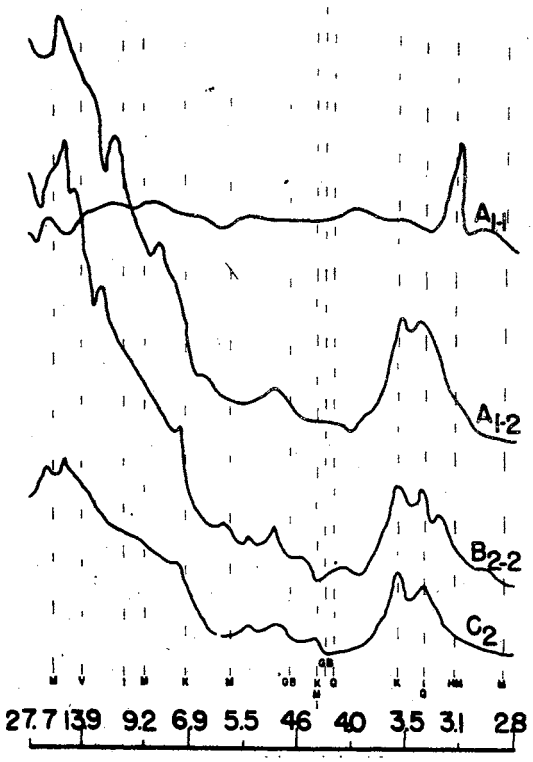
The Clay minerals shown to be dominant in the Dennis profile by X-ray analysis are montmorillonite and kaolinite. Kaolinite is shown to dominate the coarse clays and montmorillonite the fine clays. Illite is shown to be present in all horizons of the coarse clays. All horizons of the coarse clays except the B_1 contain two minerals from either the montmorillonitic, illitic or kaolinitic group.

In the fine clays, illite is shown to be absent except in the B_3 and C_1 horizons. A mixed layered grouping of montmorillonite and illite probably exists in the A_1 horizon. Small amounts of halloysite, vermiculite and hydrous micas exist in the fine clays.

Differential thermal analysis substantiates the presence of montmorillonite and kaolinite shown by X-ray analysis to be in the two size separates.



Angstroms
Fig. 3. Parsons coarse clay X-ray Spectrograph



Angstroms
Fig. 4. Parsons fine clay X-ray Spectrograph

The results of X-ray analysis in the Parsons profile show results similar to those of the Dennis profile. Montmorillonite is the dominant clay mineral present in the fine clays. Kaolinite is shown to dominate the coarse clays of all horizons except the surface where hydrous micas are dominant.

A striking similarity between the Dennis and Parsons coarse clays is the absence of montmorillonite in the B_1 of the Dennis and in the A_{2-1} and A_{2-2} of the Parsons. Other data have shown these horizons to have been subjected to intensive weathering.

Differential thermal analysis indicates the presence of kaolinite in the fine clays in horizons where it was not detected by X-ray analysis. Illite is shown to be absent in the clays of the A_{1-1}, A_{2-1}, B_{2-1} and B_{3-2} horizons of the Parsons profile.

Table XII. Clay Minerals* Found to be Present in the Two Clay Fractions of the Parsons Profile by X-ray and Differential Thermal Analysis**

Field Horizon Designation	Coarse Clay		Fine Clay	
	X-ray	Differential Thermal	X-ray	Differential Thermal
A_{1-1}	Hm(k Q M)	K	M	M K
A_{1-2}	K(Q M)	K	M (I Hm)	M K
A_{2-1}	K(I Q)	K	M (K)	M K
A_{2-2}	K(I Q)	K	M (K I)	M K
B_{2-1}	K(I Q M)	K M	M	M K
B_{2-2}	K(I M/I)	K M	M (I)	M K
B_{3-1}	K(I Q M)	K M	M (I)	M K
B_{3-2}	K(I Q M)	K M	M (Hm K)	M K
C_1	K(I Q M/I)	K	M (I K)	M K
C_2	K(I Q M)	K	M (I K)	M K

*M-Montmorillonite, K-Kaolinite, I-Illite, Q-Quartz, Hm-Hydrous micas, M/I-Mixed-Layered Minerals. Dominant minerals present are outside parenthesis. Other minerals present are enclosed by parenthesis.

**Interpretation of analysis by Dr. S. B. McCaleb. North Carolina State Experiment Station.

X-ray and differential thermal analysis of the two size fractions of the clays in both profiles show that minerals of each of the three mineral groups, montmorillonite, illite and kaolinite are present in each horizon. In horizons that contained only one or two of the minerals of the three mineral groups in one of the clay separates, a mineral of the remaining group was present in the other size separate.

Quartz, an inactive material in cation exchange relations, was shown to be present in the clays of both profiles. Whiteside and Marshall (52) interpreted an accumulation of quartz in surface horizons as an indication of weathering and loss of less resistant materials.

No quantitative measurement was made of quartz. Its presence, as indicated by X-ray analysis, suggests that it is residual.

Electron Microscope Pictures

Electron microscope pictures were made of the materials found in the two clay fractions of each horizon of the profiles examined.

The identification of clay minerals by electron microscope pictures has inherent errors. The technique of mounting specimens on a viewing screen involves drying material from a dilute suspension. The possibility of aggregation of particles upon drying cannot be avoided. Consequently, the objects seen in a picture may be either single particles or aggregates of smaller particles. Figures 5-7 are electron microscope pictures of homogeneous clay minerals that have been used in this study as reference minerals.

The light circular objects in Figure 5 are holes formed in the collodian film on drying. The dark, angular figures in all of the pictures are shadows cast by the clay mineral particle.

The characteristic shape of each mineral of the three mineral groups is shown in these figures. Particles of kaolinite exhibit sharp, distinct angles with many hexagonal particles. Montmorillonite is composed of very thin plate structures that have few distinct edges. Illite particles appear to be dense, roughly angular particles with little, if any definite configuration.

Figures 8-15 were selected as representative electron microscopic photographs of the clays found in these two profiles.

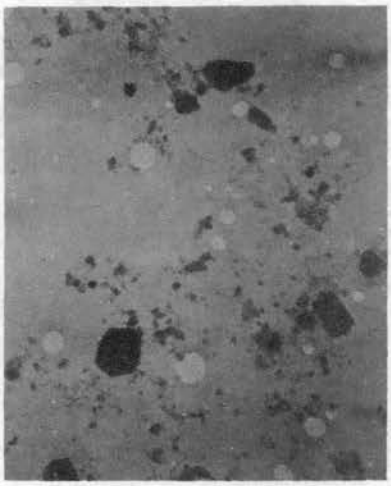


Fig. 5. Illite
coarse clay

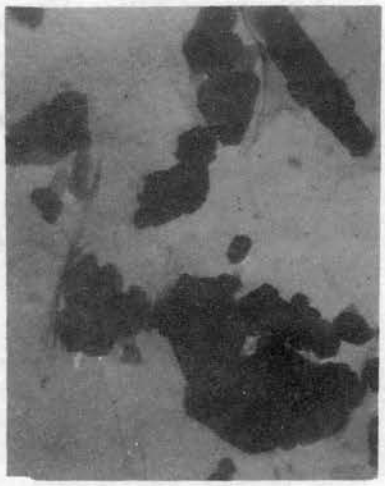


Fig. 6. Kaolinite
coarse clay

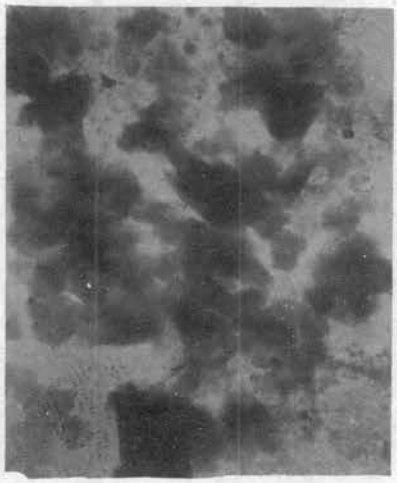


Fig. 7. Montmorillonite
coarse clay

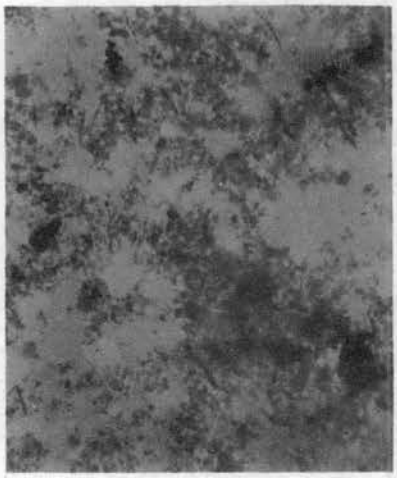


Fig. 8. Dennis A_1
fine clay

Fig. 9. Dennis A_1
coarse clay

Fig. 10. Dennis B_2
fine clay

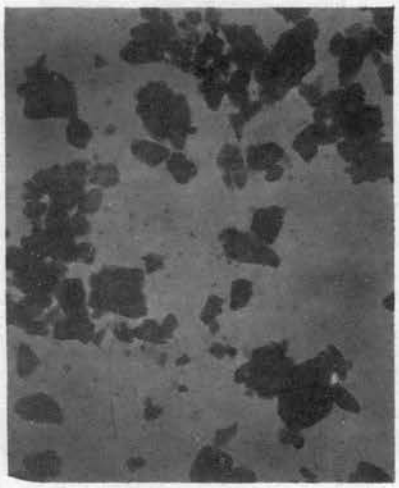


Fig. 11. Dennis B_2
coarse clay

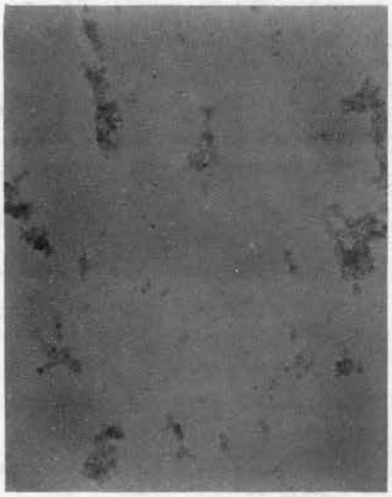


Fig. 12. Parsons A_{1-1} fine clay

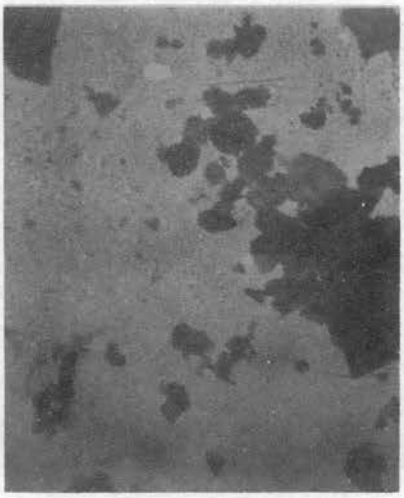


Fig. 13. Parsons A_{2-2} coarse clay

Fig. 14. Parsons B_{2-1} fine clay

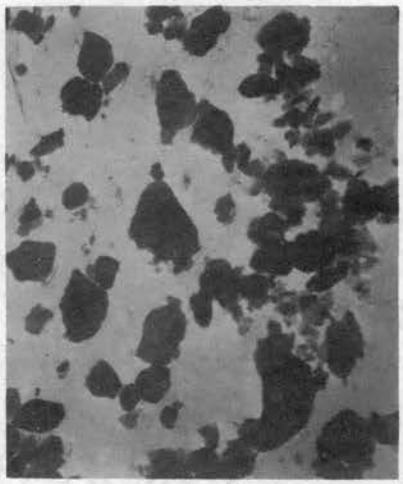


Fig. 15. Parsons B_{2-2} coarse clay

The coarse clays of both profiles are distinctly angular particles. Data in Table VII show these minerals to have a low exchange capacity. They are minerals that contain a high percentage of non-exchangeable potassium. X-ray analysis shows them to be dominantly kaolinitic with lesser amounts of other minerals present.

The fine clays of the two profiles exhibit particles with shapes similar to those of the coarse clays, but in reduced size. Many of the smaller particles are very thin and indefinite in their outline.

No distinct changes were observed by electron microscope pictures in the physical characteristics between horizons in either of the two profiles.

Quantitative Estimation of Minerals

The data from results of ethylene glycol and non-exchangeable potassium analyses have been used to estimate the relative percentages of montmorillonitic and illitic minerals present in a mixture. Data from these analyses and from cation exchange values have been used by Wilkinson and Gray (54) and Martin (39) for clay mineral estimations in similar studies.

Quantitative estimation of the amount of a specific mineral group found to be present in the two clay fractions of the horizons of the profiles studied was made by evaluation of ethylene glycol retention values for expanding lattice type clays and non-exchangeable potassium for non-expanding 2:1 lattice layered minerals.

The use of surface areas and potassium content in estimating the quanities of specific mineral groups present has numerous inherent errors.

In estimations of mineral quantities by internal surface areas, all of the internal ethylene glycol retention is credited to montmorillonitic type minerals. Other minerals are known to exhibit inter-layer expansion. Illite exhibits limited expansion and shrinkage characteristics. Vermiculite, shown to be present in some of the samples by X-ray analysis, is an expanding type mineral. Halloysite also exhibits inter-layer adsorption. No specific analysis can be made of a mixture to measure the interference these minerals have in the results of surface area determinations.

The quantity of vermiculite present, because of its expanding nature is included with the montmorillonitic type minerals.

All of the potassium found in the clays was assumed to be present in the mineral structure of illite. Illite is variable in its potassium content, Grim, Bray and Bradley (13) found it to contain from 4.7 to 6.9 percent K_2O. The sample of illite used as a reference mineral in this study contained 6.8 percent K_2O in the fine fraction and 7.5 percent in the coarse fraction. Hydrous micas, shown to be present in the profiles examined, is higher in potassium content than is illite.

In this investigation the value obtained for the internal surface areas of the two size fractions of the montmorillonite reference sample was taken to represent 100 percent montmorillonite. These two values were 504 $(m)^2$ per gram of clay in the less than .1 micron sized particles and 408 $(m)^2$ per gram clay in the 2-.1 micron sized particles.

A similar method was used to estimate the illite content. Six percent K_2O was used to represent 100 percent illite. The illite content of the sample was computed relative to this figure.

The sum of the values for montmorillonite and illite in a sample was subtracted from 100 percent. The remainder was assumed to be other minerals indicated to be present by X-ray and differential thermal analysis.

X-ray and differential thermal analysis has shown the remaining unknown portion of the sample to be dominantly kaolinite, however, quantities of quartz, and halloysite of the kaolinite mineral group is probably present in the clays of the two profiles.

In estimating the mineral content of an unknown sample by these methods, contamination by minerals that exhibit characteristics similar to the mineral group being analyzed for is probably the largest single source of error. Martin, (39) using these methods of estimation in a similar investigation, considered his values to be correct within an error of plus or minus 10 percent.

The values given in Table XIII are estimations of the amount of each mineral group found in the two size separates of the clays in the Dennis profile.

Table XIII. Estimation of the Mineral Content of the Clays in the Dennis Profile

	Minerals* <.1 Micron			Minerals 2-.1 Micron		
Horizon	M.	I.	K.Q.Etc.	M.	I.	K.Q.Etc.
	%	%	%	%	%	%
A_1	31	41	28	7	78	15
A_3	29	32	39	10	74	16
B_1	28	34	38	16	76	8
B_2	31	30	39	7	76	17
B_3	32	32	36	14	78	8
C_1	40	40	20	10	75	15
C_2	50	40	10	9	78	13
C_3	44	28	28	8	85	9

* M-Montmorillonite, I.-Illite, K.-Kaolinite, Q.-Quartz.

In the fine clays, illite and montmorillonite mineral groups are present in near equal proportions through the depth of the solum. The

montmorillonitic type with its higher exchange capacity and shrinking and swelling characteristics is dominant with the percentage that is present in the mixture.

The coarse clays are shown by the data in Table XIII to be dominantly illitic. This is in contradiction to the results of X-ray and differential thermal examinations. Illite is indicated to be present in the coarse clays of all horizons of the Dennis by X-ray analysis, but not as the dominant mineral. Differential thermal analysis did not detect the presence of illitic minerals. Grim (12) stated that X-ray analysis could be used for positive identification of illitic minerals, but that differential thermal analysis was of little value in illite determinations. The high K_2O content of the coarse clay shows this size fraction to contain a high percentage of illite.

Table XIV. Estimation of the Mineral Content of the Clays in the Parsons Profile

Horizon	Minerals* <.1 Micron			Minerals 2-.1 Micron		
	M.	I.	K.Q. Etc.	M.	I.	K.Q. Etc.
	%	%	%	%	%	%
A_{1-1}	56	27	17	5	70	25
A_{1-2}	58	30	12	7	64	29
A_{2-1}	48	28	24	11	71	18
A_{2-2}	48	26	26	17	67	16
B_{2-1}	42	27	31	28	61	11
B_{2-2}	50	27	23	11	70	19
B_{3-1}	52	27	21	12	69	19
B_{3-2}	54	30	16	2	67	31
C_1	55	29	16	9	63	28
C_2	44	52	4	8	44	48

*M.-Montmorillonite, I.-Illite, K.-Kaolinite, Q.-Quartz.

Values obtained in estimating the percentage of each mineral group present in the two clay fractions of the Parsons profile are presented in Table 14.

The fine clays of the Parsons profile are shown to be dominantly montmorillonitc. The data presented in Table XIV show the coarse clays of the Parsons profile to be dominantly illitic as were the coarse clays of the Dennis.

Mechanical analysis has shown the Parsons profile to be strongly developed and to have undergone extensive eluviation and illuviation. If selective eluviation of a specific mineral group has occurred, it should be apparent in the detailed breakdown of the profile as shown in Table XIV. The method by which the data of Tables XIII and XIV are derived eliminates the effect of normal eluviation and illuviation. Increases or decreases in a mineral group which occur in the data must necessarily be results of the effects of weathering or due to selective eluviation.

In the Parsons fine clays (Table XIV) there is an increase in kaolinite content in the A_2 and B horizons. There is a decrease in the kaolinite content in the coarse clays in these same horizons. The change in both fractions is apparent in the A_2 horizon, the horizon of maximum eluviation, and reaches its maximum in the B_{2-1}. The fact that these changes occur in the horizons which they do, suggest weathering of material from coarse to fine sized fractions.

Calculations, combining the sum of the specific clay minerals of the two size fractions of each horizon derived by ethylene glycol retention and non-exchangeable potassium values, and the mechanical analysis of the clay content in that horizon, reveals some interesting information. The result is an estimate of the amount of each mineral group present in the horizon.

The data presented in Table XV are the estimated percentages of each mineral group present in the horizons of the Dennis profile.

The illite content of the clays of the Dennis profile is a striking feature. The slightly weathered parent material is very high in illite content. As morphological processes proceeded the illite content was gradually and consistently reduced. Weathering, shown by other data is substantiated by these values.

Table XV. Estimation of Percentages of Different Clay Minerals Found in the Dennis Profile

Horizon	Clay Content	Mont-morillonite	Illite	Kaolinite Quartz, Etc.
	%	%	%	%
A_1	20	5	10	5
A_3	23	5	11	7
B_1	31	8	13	10
B_2	43	10	19	14
B_3	42	11	21	10
C_1	43	12	23	8
C_2	41	10	25	6
C_3	37	8	23	6

The increase in kaolinite content in the B horizons shows illuviation of this mineral group. Mechanical analysis has shown illuviation in the Dennis profile to be confined to the fine clays. The data suggest that kaolinitic and montmorillonitic minerals of the fine clays were illuviated.

Clay mineral weathering in the Dennis profile was most extensive in the illitic type minerals and was confined primarily to the coarse clays.

Estimations of the amounts of each clay mineral group found in the Parsons profile are given in Table XVI.

Illuviation that was evident in two of the mineral groups in the Dennis profile, Table XV, is apparent in each of the three mineral groups in the Parsons profile. Weathering that was indicated by

other data is over shadowed by the processes of eluviation and illuviation in the Parsons profile.

Table XVI. Estimation of Percentages of Different Clay Minerals Found in the Parsons Profile

Horizon	Clay Content	Mont-morillonite	Illite	Kaolinite Quartz, Etc.
	%	%	%	%
A_{1-1}	12	6	4	2
A_{1-2}	12	6	4	2
A_{2-1}	14	4	7	3
A_{2-2}	26	11	8	7
B_{2-1}	58	22	20	16
B_{2-2}	59	25	22	12
B_{3-1}	49	23	17	9
B_{3-2}	38	18	13	7
C_1	36	18	12	6
C_2	40	16	20	4

The very high montmorillonite content of the Parsons fine clays which was apparent in Table XIV, loses much of its significance when the fine clays are combined with the coarse clays.

SUMMARY AND CONCLUSIONS

Two soils of extensive area in eastern Oklahoma were examined to determine the type and amount of each clay mineral group present in the clay sized fraction of each horizon.

The Dennis and Parsons series were sampled by horizon and sub-horizon to the depth of the slightly weathered parent material. The clay portion was separated from a sample of each horizon by sedimentation. This fraction was further fractionated by use of a Sharples supercentrifuge into the size groupings of particles with diameters of 2-.1 micron and particles less than .1 micron in diameter.

The materials found in these two fractions were examined by chemical analysis, X-ray and differential thermal techniques, and with the electron microscope. From the results of these examinations, the clay mineral groups present in each of the two size separates of the clay fraction in each horizon were determined and the amount of the clay mineral groups present estimated.

The Dennis profile was found to be dominantly illitic with a high percentage of montmorillonite present. The dominant mineral in the Parsons profile was found to be montmorillonite with a high percentage of illite present. Kaolinitic type minerals were present in both profiles.

The clay fractions of both profiles contained high percentages of particles less than .1 micron in diameter. The coarse clay content of the surface horizons of both profiles was minor. The lower horizons

of the Dennis profile contained high percentages of coarse clay sized materials.

Weathering in the two profiles was noted by the change in total surface area of the clay sized particles. The loss of coarse clay content in the upper horizons of the Dennis profile was attributed to weathering. Estimation of the amounts of specific mineral groups present showed the coarse clays of the Dennis to be dominantly illite. Weathering in the coarse clays of this profile was extensive in the illitic type minerals.

The possibility of selective eluviation of specific mineral groups in profile weathering was investigated. All minerals were present in the illuviated material in a ratio similar to their occurance in the clays of the topsoil.

Eluviation in the Dennis profile was primarily confined to the particles of less than .1 micron in diameter. In the Parsons profile, both size fractions were eluviated. Although a large volume of the illuviated material was composed of particles less than .1 micron in diameter, these materials were not selectively eluviated.

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